AMENDMENT NO. 3 SEPTEMBER 2018
TO
IS 13704 : 1993 GLYCEROL ESTERS OF WOOD ROSIN (ESTER GUMS), FOOD GRADE — SPECIFICATION

(*Page* 1, *clause* **3.2.1**) — Insert the following new clauses after **3.2.1**:

'**3.2.2** *Specific Gravity*

The specific gravity of 50 percent solution in d-limonene of the material at 20°/25°C shall not be less than 0.935.

3.2.3 *Gas Chromatography of Resin Alcohols and Glycerol*

When tested as per the method given at Annex C of this standard, the sample shall pass the test.

[*Page* 2, *Table* 1, *Sl No*. v), *col* 3 (*see also Amendment No. 1*)] — Substitute '1' *for* '2'.'

(*Page* 4, *clause* **B-3.2.1**) — Insert the following new annex at the end of the clause:

ANNEX C
(*Clause* 3.2.3)

GAS CHROMATOGRAPHY OF RESIN ALCOHOLS AND GLYCEROL

C-1 PRINCIPLE OF THE METHOD

The complex ester groups in the glycerol esters of wood rosin are reduced by reaction with a metal hydride (sodium bis (2-methoxy-ethoxy) aluminium dihydride) in toluene solution to form a mixture of resin alcohols and glycerol. Excess reagent is then hydrolyzed with aqueous acid forming two phases. Gas chromatography of the toluene phase produces a characteristic chromatogram of the resin alcohols that abietyl and dehydroabietyl alcohols are predominates. Confirm the presence of two predominate peaks of both resin alcohols with the typical gas chromatogram as fiven in Fig 3. Chromatography of the neutralized aqueous phase on a different column verifies the presence of glycerol.

C-2 APPARATUS

C-2.1 Gas Chromatograph, equipped with a flame ionization detector. Recorder: 0 to 1 V

C-2.2 Analytical Balance, capable of weighing to the nearest 0.001 g.

C-2.3 Centrifuge, table top, capable of achieving 3 200 rpm.

C-3 REAGENTS

C-3.1 Toluene, reagent grade.

C-3.2 Sodium bis (2-methoxyethoxy) Aluminium Dihydride, 70 percent in toluene. Pipette 10.0 ml into a 100 ml volumetric flask. Dilute to volume with toluene and mix thoroughly.

C-3.3 Hydrolysis Solution — Slowly add 50 ml of concentrated sulphuric acid, reagent grade, to 200 ml distilled water while stirring in an ice bath. Cool to room temperature.

C-3.4 Phenolphthalein Solution — 1 percent in ethanol.

C-3.5 Sodium Hydroxide Solution: Dissolve 16 g of reagent grade NaOH in 70- 80 ml of distilled water and cool to room temperature. Dilute to 100 ml with distilled water and mix thoroughly. Store it in a polyethylene bottle.

C-3.6 1,4-Butanediol of known purity, above 99 percent.

C-3.7 Glycerol of Known Purity, above 99 percent.

C-3.8 Internal Standard Solution, weigh 0.1 g of 1,4-butanediol into a 100 ml volumetric flask. Dilute to volume with distilled water and mix thoroughly. Glycerol Solution: weigh 0.1 g of 1,4-butanediol and 0.1 g glycerol into a 100 ml volumetric flask. Dilute to volume with distilled water and mix thoroughly

C-4 PROCEDURE

C-4.1 Procedure for Resin alcohols

C-4.1.1 Weigh 250-300 mg sample into a 25 ml Erlenmeyer flask containing a stirring bar (Teflon coated, 1 inch). Pipette 5.0 ml toluene into the flask and stir magnetically until sample is dissolved. Pipette 5.0 ml of sodium bis (2-methoxyethoxy) aluminium dihydride Reagent into the flask, cap, and stir for 30 min.

C-4.1.2 Uncap and, while stirring, pipette 3.0 ml of hydrolysis solution into the flask. Continue stirring for 3 min. Transfer contents of flask to centrifuge tube (15 ml), stopper, and shake vigorously. Vent and centrifuge at 2 800 - 3 200 rpm for 5 min.

C-4.1.3 Inject 0.5 µl of the upper layer into the gas chromatograph operating under the specified conditions and record the chromatogram. Compare with the chromatograms shown below to verify the approximate retention order of the resin alcohols.

C-4.1.4 Chromatographic conditions during the test shall be as follows:

a) *Column* — DB-1 methyl silicone (bonded and crosslinked), 15 m, widebore capillary (0.53 mm ID), film thickness 1.5 µm
b) *Temperature range* — 60°C to 300/320°C. A direct flash vaporization injection port liner is recommended.
c) *Flow rates* — 1) Carrier Gas (Helium) : 30 ml/min at 4.4 to 4.5 kg/cm^2
    2) Hydrogen : 30 ml/min
    3) Air : 240 ml/min
d) *Temperatures* — 1) Column : Isothermal at 190°C,
    2) Inlet : 250°C
    3) Detector : 250°C

C-4.2 Procedure for Glycerol

C-4.2.1 Using a pipette or hypodermic syringe, remove the toluene layer and part of the aqueous layer leaving approximately 2 ml of the aqueous layer in the centrifuge tube.

C-4.2.2 Add 1 drop of phenolphthalein solution and neutralize with the sodium hydroxide solution. Aluminium salts will precipitate.

C-4.2.3 Pipette 5 ml of the internal standard solution into the tube, dilute to 15 ml with distilled water, stopper, shake, and then centrifuge at 2 800 – 3 200 rpm for 5 min.

C-4.2.4 Inject 1 µl of the clear supernatant liquid into the gas chromatograph operating under the specified conditions and record the chromatogram.

C-4.2.5 Inject 1 µl of the glycerol solution and record the chromatogram. Measure the retention times of any observed peaks relative to 1,4- butanediol. Compare retention times to that of glycerol.

C-4.2.6 Chromatographic conditions during the test shall be as follows:

a) Column: DB-WAX polyethylene glycol (bonded and cross-linked), 15 m, wide bore capillary (0.53 mm ID), film thickness 1.0 µm
b) Temperature range: 20°C to 230°C.
c) Flow rates:
    1) Carrier gas (Helium) : 30 ml/min at 4.3 kg/cm^2
    2) Hydrogen : 30 ml/min
    3) Air : 240 ml/min
d) Temperatures:
    1) Column : Programmed at 120 to 200°C at 6°C/min,
    2) Inlet : 250°C
    3) Detector : 250°C

(FAD 08)

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